

## SEARCH REQUEST FORM

Scientific and Technical Information Center

Requester's Full Name: Jeffrey E. Russel Examiner #: 62785 Date: 12-4-2002  
 Art Unit: 1654 Phone Number 30 8-3975 Serial Number: 09/815978  
 Mail Box and Bldg/Room Location: \_\_\_\_\_ Results Format Preferred (circle) PAPER DISK E-MAIL

CMI-11D13/CMI 9807

If more than one search is submitted, please prioritize searches in order of need.

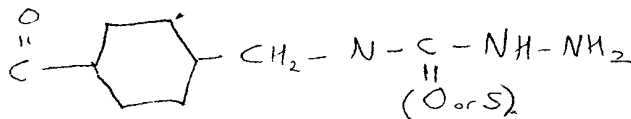
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Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc, if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

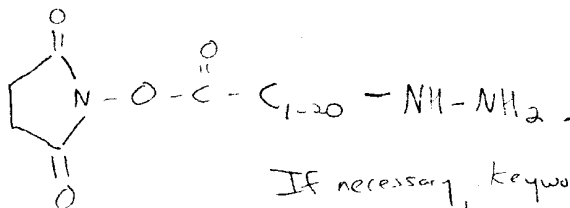
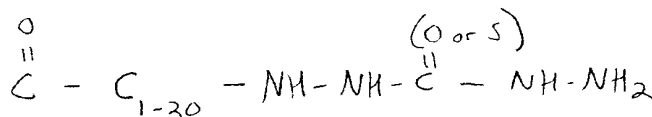
Title of Invention: Hydrazine-Based And Carbonyl-Based Bifunctional Crosslinking ReagentsInventors (please provide full names): D. SchwartzEarliest Priority Filing Date: 3-22-2001

\*For Sequence Searches Only\* Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.

Please search the following partial structure:



Cyclohexyl group.



If necessary, keywords are conjugat?, crosslink?,  
 bifunctional, antibody, immobili?. Thank you.

Edward Hart  
 Technical Info. Specialist  
 STIC/Biotech  
 CMI 6B02 Tel: 305-9203

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## STAFF USE ONLY

## Type of Search

## Vendors and cost where applicable

Searcher: \_\_\_\_\_ NA Sequence (#) 1 STN  
 Searcher Phone #: \_\_\_\_\_ AA Sequence (#) \_\_\_\_\_ Dialog \_\_\_\_\_  
 Searcher Location: \_\_\_\_\_ Structure (#) \_\_\_\_\_ Questel/Orbit \_\_\_\_\_  
 Date Searcher Picked Up: 12/6/02 Bibliographic \_\_\_\_\_ Dr. Link \_\_\_\_\_  
 Date Completed: 12/12/02 Litigation \_\_\_\_\_ Lexis/Nexis \_\_\_\_\_  
 Searcher Prep & Review Time: \_\_\_\_\_ Fulltext \_\_\_\_\_ Sequence Systems \_\_\_\_\_  
 Clerical Prep Time: \_\_\_\_\_ Patent Family \_\_\_\_\_ WWW/Internet \_\_\_\_\_  
 Online Time: \_\_\_\_\_ Other \_\_\_\_\_ Other (specify) \_\_\_\_\_

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 FILE LAST UPDATED: 11 Dec 2002 (20021211/ED)

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=> d stat que

L1 STR

12  
 31

2 7  
 C 3 C N C N N  
 1 C C 3 9 10 11

6 C C<sub>4</sub>  
 C C C  
 14 13 5

VAR G1=O/S

NODE ATTRIBUTES:

DEFAULT MLEVEL IS ATOM

DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:

RING(S) ARE ISOLATED OR EMBEDDED

NUMBER OF NODES IS 14

STEREO ATTRIBUTES: NONE

L3 135 SEA FILE=REGISTRY SSS FUL L1

L4 STR

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 G1

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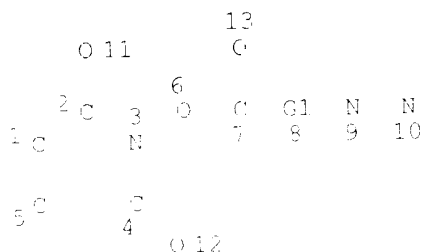
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REF G2=[1-20] C

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 DEFAULT ELEVEL IS LIMITED

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STEREO ATTRIBUTES: NONE  
 L6 78 SEA FILE=REGISTRY SSS FUL L4  
 L9 STR



REF G1=(1-20) C  
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 DEFAULT MLEVEL IS ATOM  
 DEFAULT ELEVEL IS LIMITED

GRAPH ATTRIBUTES:  
 RING(S) ARE ISOLATED OR EMBEDDED  
 NUMBER OF NODES IS 13

STEREO ATTRIBUTES: NONE  
 L11 33 SEA FILE=REGISTRY SSS FUL L9  
 L12 52 SEA FILE=HCAPLUS ABB=CN PLU=ON L3  
 L13 37 SEA FILE=HCAPLUS ABB=CN PLU=ON L6  
 L14 44 SEA FILE=HCAPLUS ABB=CN PLU=ON L11  
 L15 3 SEA FILE=HCAPLUS ABB=CN PLU=CN L12 AND (CONUGAT? OR CROSSLINK  
 1 OF BIFUNCTIONAL? OR ANITBOD? OR AB# OR MAB# OR PAB# OR  
 IMMOBILI?)  
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 1 OF BIFUNCTIONAL? OR ANITBOD? OR AB# OR MAB# OR PAB# OR  
 IMMOBILI?)  
 L18 3 SEA FILE=HCAPLUS ABB=ON PLU=ON L14 AND (CONUGAT? OR CROSSLINK  
 2 OF BIFUNCTIONAL? OR ANITBOD? OR AB# OR MAB# OR PAB# OR  
 IMMOBILI?)  
 L19 11 SEA FILE=HCAPLUS ABB=ON PLU=ON L16 OF L17 OF L18

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L19 ANSWER 1 OF 10 HCAPLUS COPYRIGHT 2002 ACS  
 ACCESSION NUMBER: 2001:713305 HCAPLUS  
 DOCUMENT NUMBER: 145:272864  
 TITLE: Hydrazine-based and carbonyl-based  
**bifunctional crosslinking** reagents  
 for biomolecules, drugs, and synthetic polymers  
 INVENTOR(S): Schwartz, David A.  
 PATENT ASSIGNEE(S): Solulink, Inc., USA  
 SOURCE: PCT Int. Appl., 27 pp.  
 CODEN: PINXD2  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 2

## PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001070685	A2	20011917	WO 2001-UN9252	20010301
W: AE, AG, AL, AM, AT, A, AZ, BA, BB, BC, BF, BY, BE, BR, CH, CL, CO, CR, CU, CZ, DE, DF, DM, DZ, EE, ES, FI, GB, GD, GE, GR, HK, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, ME, MK, MN, MW, MX, MZ, NG, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AG, BY, BG, KZ, MD, RU, SK, TR RW: GH, GN, HE, LS, MW, MD, SD, SL, SE, TZ, CG, ZM, AT, BE, BR, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SK, TR, RU, RJ, CH, CG, CI, CN, GA, GN, GW, ML, MR, NE, SN, TD, TG				
US 2002146504	A1	20021010	US 2002-51277	20020110
PRIORITY APPLN. INFO.:			US 2000-191186P	P 20000322
			US 2001-262094P	P 20010116

OTHER SOURCE(S): MARPAT 134:272864

AB Reagents and methods are provided for **bifunctional crosslinking and immobilizing** biomols., drugs, and synthetic polymers. The reagents of formula  $\text{BRANHNH}_2\bullet\text{HX}$  [wherein A =  $\text{NHCO}$ ,  $\text{NHCS}$ ,  $\text{NHNHCO}$ ,  $\text{NHNHCS}$ , or a direct bond; B = an amino or thio reactive moiety; R = specified aliphatic divalent groups containing any combination of cycloalkylene, C-R10(2), CR10:CR10, C:CR12R13, CR12R13, C:tpbond:C, O, SGA, NR10, N-R12R13, CL, etc.; a = 0-2; b = 0-3; G = O or NR10; L = S, O, or NR10; R10 = specified monovalent groups; R12 and R13 = independently H, cycloalkyl, alkenyl, alkynyl, or (hetero)aryl; or R12 and R13 together form cycloalkylene or alkenylene; X = neg. counterion; or a derivative thereof] possess a thiol or amino reactive group and a hydrazine or oxyamine moiety. Conjugates and **immobilized** biomols. are also provided. For example, hydrazinonicotinic acid was converted to the acetone hydrazone and treated with N-hydroxysuccinimide to give the **crosslinking agent**, succinimidy 6-hydrazinonicotinate acetone hydrazone (I), in 33% yield. A solution of ovalbumin in PBS and EDTA was added to a solution of I in DMF and the mixture incubated at room temperature for 4 h to afford the hydrazine-modified protein, which exhibited a molar extinction coefficient of 22,000 at 365 nm.

17 362522-51-8P

EL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(**crosslinking agent**; preparation of hydrazine- and carbonyl-based **bifunctional crosslinking agents** and use with biomols., drugs, and synthetic polymers)

L19 ANSWER 2 OF 10 HCAPLUS COPYRIGHT 2002 ACS

ACCESSION NUMBER: 1993:517414 HCAPLUS

DOCUMENT NUMBER: 119:217414

TITLE: peptide aldehyde analogs for trypsin inhibitors

INVENTOR(S): Brunck, Terence Kevin; Pepe, Michael Gary; Pearson, Daniel Andrew; Webb, Thomas Roy

PATENT ASSIGNEE(S): Corvas International, Inc., USA

SOURCE: PCT Int. Appl., Cl. 35.

CODEN: PEXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY APP. NUM. COUNT: 2

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9314779	A1	19930805	WO 1993-US906	19930129
W: CA, JP				
RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PL, SE				

EP 627925 A1 19941214 EP 1993-200176 1993-12-14  
 R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LI, NL, PT, SE, SI, TR  
 JP 07503715 TE 19930423 JP 1993-11999 1993-11-23  
 US 0684418 A 19960709 US 1993-11866 1993-11-23  
 WO 1993-028122 1993-02-12  
 WO 1993-03066 1993-03-12  
 WO 1993-03066 1993-03-12

OTHER SOURCE(S): MARPAT 119:217:14

AB Peptide aldehyde analogs are disclosed which have substantial potency and specificity as inhibitors of mammalian pancreatic trypsin. The compds. of the invention are useful in the prevention and treatment of tissue damage or destruction associated with pancreatitis. Preparation of the analogs is described. Thus, N-t-butoxycarbonyl-L-Asp-L-Pro-L-argininal (I) (preparation given) had a  $K_i$  against trypsin of 0.00045  $\mu$ M. The effectiveness of I in an animal model for pancreatitis was also demonstrated.

IT 139976-30-OP

RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation and immobilization of, in peptide aldehyde analog  
 preparation for trypsin inhibitor)

IT 139976-26-4P 139976-27-5P 139976-29-7P

139976-30-ODP, solid phase-immobilized

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT  
 (Reactant or reagent)

(preparation and reaction of, in peptide aldehyde analog preparation for  
 trypsin inhibitor)

L19 ANSWER 3 OF 10 HCAPLUS COPYRIGHT 2002 ACS

ACCESSION NUMBER: 1992:612932 HCAPLUS

DOCUMENT NUMBER: 117:212932

TITLE: Total synthesis and absolute configuration of  
 benzamide A

AUTHOR(S): Chiba, Noritaka; Tobe, Takahiko; Okada, Shinsuke;  
 Igawa, Seishiro

CORPORATE SOURCE: Fac. Sci. Technol., Keio Univ., Yokohama, 223, Japan

SOURCE: Journal of the Chemical Society, Chemical  
 Communications (1992), (15), 1064-6

CIDEN: JCCCAT; ISSN: 0022-4936

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 117:412932

GI

\* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT \*

AB The first total synthesis of the novel marine natural product, benzamide A (I) is described, revealing the **absolute** configuration of this compound. I was prepared in several steps from known ester II (R<sup>1</sup> = H-3,0<sup>2</sup>), which can be obtained from L-glutamic acid in 4 steps. Key steps were the cyclization of active ester III to give hexahydro-2-azepinone IV (R<sup>1</sup> = CH<sub>2</sub>Ph, R<sup>2</sup> = Boc) and the coupling of IV-3F3CO<sub>2</sub>H (R<sup>1</sup> = R<sup>2</sup> = H) with polyhydroxylated C10 side chain V by (EtO)2P(O)CN to give the corresponding amide.

IT 144090-64-2P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT  
 (Reactant or reagent)  
 (preparation and cyclization of)

L19 ANSWER 4 OF 10 HCAPLUS COPYRIGHT 2002 ACS

ACCESSION NUMBER: 1991:425407 HCAPLUS

DOCUMENT NUMBER: 115:25407  
 TITLE: Novel trifunctional carrier molecule for the fluorescent labeling of haptens  
 AUTHOR(S): Bredemist, Reinhard; Wexhoff, Gregory A.; Kusterbeck, Anne M.; Charles, Paul L.; Thompson, Richard K.; Ligler, Frances S.; Vogel, Carl Wilhelm  
 CORPORATE SOURCE: Dep. Biochem. Mol. Biol., Georgetown Univ., Washington, DC, 20057, USA  
 SOURCE: Analytical Biochemistry (1991), 190:2, 254-261  
 CODEN: ANBCA2; ISSN: 0003-2697  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 AB The authors developed a novel trifunctional carrier mol. for the synthesis of hapten-fluorophore conjugates as reporter mols. in immunoassays. This carrier eliminates some of the disadvantages associated with currently used fluorophore-labeling procedures including high nonspecific binding. The backbone of the carrier consists of the 21 amino acid residues of the insulin A-chain mol. This polypeptide provides a single site (terminal amino group) for covalent coupling of the hapten, three carboxyl groups for the attachment of fluorophores, and four sulfhydryl groups for derivatization with hydrophilic residues to compensate for the hydrophobic effect of the attached fluorophores. The sites for fluorophore attachment are 4, 17, and 21 amino acids away from the hapten attachment site. This spatial separation minimizes quenching of the fluorescence signal due to interaction of the fluorophores with each other and with the attached hapten. 2,4-Dinitrophenyl (DNP) was selected as model hapten, fluorescein as label, and S-sulfonate groups as hydrophilic residues. The properties of the DNP-insulin A-chain-fluorescein conjugate (DNP-Ins-Fl) were compared to those of a DNP derivative labeled with a single fluorescein moiety via a small lysine spacer (DNP-Lys-Fl). The DNP-Ins-Fl conjugate exhibited a 3-fold lower nonspecific adsorption to immobilized non-immune Ig compared to an approx. 3-fold more efficient displacement from the binding sites of an immobilized anti-DNP antibody by the antigen DNP-lysine. Furthermore, at equimolar concns. the DNP-Ins-Fl generated a 2.6-fold higher fluorescent signal than DNP-Lys-Fl. Due to these properties of DNP-Ins-Fl, DNP-lysine could be detected with an approx. 10-fold higher sensitivity compared to DNP-Lys-Fl as labeled antigen. The use of DNP-Ins-Fl as reporter molecule in a competitive fluorimmunoassay allowed the quant. determination of picomole amts. of DNP-lysine.  
 IT 134664-50-9  
 FL: RCT (Reactant); FACT (Reactant or reagent)  
 (reaction of, with FITC)

L19 ANSWER 5 OF 10 HCAPLUS COPYRIGHT 2002 ACS  
 ACCESSION NUMBER: 1990:4 2:31 HCAPLUS  
 DOCUMENT NUMBER: 115:261  
 TITLE: Preparation and characterization of immunoconjugates for antibody-targeted photolysis  
 AUTHOR(S): Fekete, S.; Tompkins, Ronald G.; Yarnish, Martin L.  
 CORPORATE SOURCE: Cent. Adv. Biotech. Med., Rutgers, State Univ., Piscataway, NJ, 08855, USA  
 SOURCE: Bioconjugate Chemistry (1991), 1:1, 212-21  
 CODEN: BICHE3; ISSN: 1043-1802  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 AB Monoclonal antibody (MAB)-dextran-tin(IV) chlorin e6(SnCe6) immunoconjugates were prepared by a new technique involving the use of reducing terminal-modified dextran carriers and site-specific modification of the Fc oligosaccharide moiety on the antibodies. Dextran carriers were synthesized to increase the number of SnCe6 mols. attached to a MAB

The dextran carriers were coupled to the MAb via a hydrazide, chain-terminal hydrazide group to prevent conjugation of MAbs. Conjugates were prepared with anti-melanoma MAb 2.1 containing 12.9 SnCe6 mols. per MAb. Under neutral conditions, no hydrolysis of the hydrazide bond between the MAb and the dextran carrier could be detected, and the hydrazide was not stabilized by reaction with  $\text{H}_2\text{NBNH}_3$  or  $\text{NaBH}_4$ . Anal. of the purified immunconjugates showed that approx. 2 dextran carrier chains were attached to a MAb regardless of the number of SnCe6 mols. linked to a dextran carrier. Site-specific covalent attachment of the SnCe6-dextran chains to the MAb was confirmed by SDS-PAGE. HPLC anal. of the conjugates gave a single species eluting in the range of 200-240 kDa. As determined by a competitive inhibition RIA using viable SK-MEL-2 human malignant melanoma cells, the conjugates showed excellent retention of antigen-binding activity relative to unconjugated MAb.

IT 127381-73-1P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation and hydrazinodextran terminal hydrazide protection by)

L19 ANSWER 6 OF 10 HCAPLUS COPYRIGHT 2002 ACS

ACCESSION NUMBER: 1974:13710 HCAPLUS  
DOCUMENT NUMBER: 40:13710  
TITLE: Production of a foam material  
INVENTOR(S): Ouchi, Naoshi; Nakamura, Toshiro  
PATENT ASSIGNEE(S): Unisika Co., Ltd.  
SOURCE: Jpn. Tokyoko Koho, 2 pp.  
COLLEN: JAXXAD  
DOCUMENT TYPE: Patent  
LANGUAGE: Japanese  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 47181944	B4	1973-12-27	JP 1969-88917	1969-11-06

AB (α-Acetylethylidene) carbonylhydrazide (I) [ 50883-75-5]  
 $(\text{CH}_3\text{COOC}(\text{ME})\text{:NNHCONHNH}_2$ , which generated nontoxic, odorless, nonflammable gas on decomposition was used as a blowing agent for manufacture of polymer foams.

Thus, 93 parts ABS copolymer [9003-56-9] was dry-blended with 1 parts I and injection molded at die temperature 200 deg. at 47 mm/sec. to give foam having uniform small cells and an apparent sp.gr. 0.144 g/cm<sup>3</sup>.

IT 50883-75-5

RL: USES (Uses)  
blowing agents, for manufacture of polymer foams)

L19 ANSWER 7 OF 10 HCAPLUS COPYRIGHT 2002 ACS

ACCESSION NUMBER: 1967:75307 HCAPLUS  
DOCUMENT NUMBER: 40:75307  
TITLE: Preparation of terephthaloyl diisocyanate  
AUTHOR(S): Neidlein, Richard; Bottler, Rainer  
CORPORATE SOURCE: Univ. Marburg-Lahn, Marburg-Lahn, Ger.  
SOURCE: Chem. Ber. (1967), 100(2), 698-700  
COLLEN: CHBEAM  
DOCUMENT TYPE: Journal  
LANGUAGE: German

GI For diagram(s), see printed CA Issue.

AB p-C6H4(CONH<sub>2</sub>)<sub>2</sub> (32.8 g.) in 200 cc. dry CHCl<sub>3</sub> refluxed about 10 days with 153.4 g. (COCl)<sub>2</sub> gave 45.2 g. p-C6H4(CONCO<sub>2</sub>)<sub>2</sub> (I), m. 111-112°. (2.7 g.) in 60 cc. tetrahydrofuran treated with cooling with 1.0 g. absolute MeOH and stirred 1 hr. at room temperature yielded 0.9 g. p-C6H4(CONHCO<sub>2</sub>R)<sub>2</sub> (II) (R = Me). Similarly prepared were the III with R, m.p. (decomposition), and IV given: Et, 147-148°, 0.7 g.;

208-9°, 95; MeOCH<sub>2</sub>CH<sub>2</sub>, 159-61°, 92; Ph, 178-9°, 67.  
Similarly prepared were p-CH<sub>3</sub>(CONHCOX)<sub>2</sub> (III) (X = SPH), m. 124-5° (decomposition), 89; III (X = NHCH<sub>2</sub>CH<sub>2</sub>(OEt)<sub>2</sub>), 85, m. 227-8° (decomposition), from MeOCH<sub>2</sub>CH<sub>2</sub>OH; and III (X = NHNHPh), 87, m. above 360° (PhCN). I (3.64 g.) in 50 cc. dry tetrahydrofuran treated dropwise with stirring with 17.1 cc. 8.5% dry HNO<sub>3</sub>-CH<sub>2</sub>Cl gave 4 g. III (X = N<sub>2</sub>), m. 234-5°. I (2.4 g.) in 50 cc. dry tetrahydrofuran treated with stirring and cooling with 1 equivalent 6.2% CH<sub>2</sub>Cl-Ph<sub>3</sub> gave 4 g. III, m. 252-3° (MeOCH<sub>2</sub>CH<sub>2</sub>OH).

IT 13506-12-2P 14994-19-5P

RI: SPN (Synthetic preparation); PNP Preparation, (preparation of)

L19 ANSWER 8 OF 10 HCAPLUS COPYRIGHT 2002 ACS

ACCESSION NUMBER: 1967:5072- HCAPLUS

DOCUMENT NUMBER: 60:557L

TITLE: Hydrazine compounds as heteroconstituents in peptides.  
VII. Synthesis of derivatives and peptides of  
DL-α-hydrazino-β-phenylpropionic acid  
(NHPhe).

AUTHOR(S): Gupta, Kanate; Friedrich, Hartmut  
CORPORATE SOURCE: Deut. Acad. Wiss., Berlin, Ger.  
SOURCE: Chem. Ber., 1968, 99(12), 3914-24  
DESEN: CHBEAM

DOCUMENT TYPE: Journal

LANGUAGE: German

AB cf. CA 64, 307c. The following abbreviations are used: NHPhe = α-hydrazino-β-phenylpropionic acid or -propionyl; NH<sub>2</sub>ly = hydrazinoacetic acid or -acetyl; BOC = tert-BuO<sub>2</sub>C; Z = PhCH<sub>2</sub>CO<sub>2</sub>C; OSu = diisopropylcarbodiimide; Et = Et ester; OMe = Me ester; OSu = hydroxysuccinimide ester; ONP = p-nitrophenyl ester; THF = tetrahydrofuran; DMF = N,N-dimethylformamide. To 3.3 g. DL-NHPhe·HCl (VA 62, 1961) in 40 cc. absolute EtOH was added 5.4 cc. Et<sub>3</sub>N and the product heated with 7.5 g. BOC-N<sub>2</sub> 1 hr. at 35° to give 1.3 g. II. DL-NHNHCH(CH<sub>2</sub>Ph)CONH<sub>2</sub> (I, R = BOC, X = OH) (II). Free NHPhe (2.44 g.) in 10 cc. EtOAc let stand 24 hrs. at room temperature with 1.57 g. BOC-N<sub>2</sub> gave 1.4 g. II. II (1.54 g.) in 20 cc. MeOH treated during 30 min. with 10 cc. N NaOH gave 1.3 g. I (R = BOC, X = OH) (III). A solution of 1.80 g. NHPhe in 6 cc. 2N NaOH and 10 cc. dioxane stirred 24 hrs. at 35° with 1.57 g. BOC-N<sub>2</sub> and 0.05 g. MgO gave 1.3 g. III. II (1.54 g.) in 10 cc. saturated MeOH-NH<sub>3</sub> let stand 2 days, with 100 mg. 1,2,4-triazole gave 0.43 g. I (R = BOC, X = N<sub>2</sub>H<sub>3</sub>) (IV). To 1.54 g. II in 10 cc. absolute MeOH were added 101 mg. 1,2,4-triazole and 1 cc. 100% N<sub>2</sub>H<sub>4</sub>·H<sub>2</sub>O and, after 2 days the MeOH was evaporated to give 1.35 g. I (R = BOC, X = N<sub>2</sub>H<sub>3</sub>) (V). To 33 g. PhCH<sub>2</sub>CO<sub>2</sub>H in 125 cc. absolute CH<sub>2</sub>Cl was added 61 g. ClCO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NC<sub>2</sub>-p at 0-5° with stirring and the reaction solution stirred 3 hrs. at room temperature to give 35.3 g. Z-ONP. Free NHPhe-OEt from 2.09 g. HCl salt treated with 2.73 g. Z-ONP like II (BOC-ONP procedure) gave 3.2 g. crude I (R = Z, X = OEt) (VI), decomposing on distillation. Crude VI (1.71 g.) saponified like

III (MeOH-aqueous NaOH method) gave 1.35 g. I (R = Z, X = OH) (VII). Crude VII (1.5 g.) in 12.5 cc. 2N NaOH treated during 2 hrs. with 1.57 g. BOC-N<sub>2</sub> and 12.5 cc. 2N NaOH with ice cooling (the pH was kept at 11), and the mixture stirred 30 min. gave 4.0 g. VIII. VI (1 g.) treated with saturated MeOH-NH<sub>3</sub> and 1,2,4-triazole like IV gave 1.5 g. I (R = Z, X = NH<sub>2</sub>) (IX). IX (1.5 g.) treated like V gave 1.5 g. I (R = Z, X = N<sub>2</sub>H<sub>3</sub>). Free DL-NHPhe·HCl from 3.03 g. HCl salt kept 30 min. in 15 cc. Me<sub>2</sub>CO gave 2.6 g. DL-Me<sub>2</sub>CO·NHNHCH(CH<sub>2</sub>Ph)CONH<sub>2</sub> (III) (1.4 g.) in 12 cc. DMF treated with 0.58 g. N-hydroxysuccinimide VIII, and then at 0° with 1.03 g. DCCl and the reaction mixture kept 60 hrs. at 0° gave 1.5 g. I (R = BOC, X = OSu) (VIIIa). VII (3.14 g.) and 1.15 g. VIII in 15 cc. THF treated with 2.06 g. DCCl 24 hrs. at 0° gave 0.4 g. I (R = Z, X = OSu). DL-NHPhe-OEt·HCl (1.83 g.) suspended in 14 cc. THF, treated with 1.78 g.



Et<sub>3</sub>N with ice cooling, the product treated with 1.5 cc. Z-Gly and then with 1.52 g. DCCl with ice cooling, and the mixture kept overnight at 0° gave 1.66 g. NP-Z-Gly-L-NHPh-*R* (IX) (*R* = OH). XI (1.8 g.) from 2 g. HCl salt) in 30 cc. EtOAc kept 24 hrs. at room temperature with 0.2 g. Z-Gly-OCH<sub>2</sub>CO<sub>2</sub>N gave 9.1 g. X, m. 86-87°. X (2 g.) saponified like III gave 1.5 g. IX (*R* = OH). X (3.99 g.) in 10 cc. **absolute** MeOH kept 3 days with 2.5 cc. 100% N<sub>2</sub>H<sub>4</sub>·H<sub>2</sub>O and approx. 100 mg. 1,2,4-triazole gave 2 g. IX (*R* = N<sub>2</sub>H<sub>3</sub>). Free DL-NHPh-*OEt* (from 2.4 g. HCl salt) and 2.1 g. Z-L-Ala in 30 cc. MeCN treated portionwise with 2.46 g. DCCl 24 hrs. at 0°, 2 drops AcOH added, and the mixture let stand 2 hrs., gave 2.4 g. NP-Z-L-Ala-DL-NHPh-*R* (XII) (*R* = OH). XII (1.03 g.) in 5 cc. CHCl<sub>3</sub> combined with 3.44 g. Z-L-Ala-ONP in 6 cc. CHCl<sub>3</sub>, 0.05 cc. AcOH added, and the solution kept 48 hrs. at room temperature gave 3.8 g. XII. XII (1.03 g.) saponified like III (MeOH-aqueous

NaOH

method) gave 0.62 g. XI (*R* = OH). XII (1.03 g.) in 8.4 cc. **absolute** AcOH heated 40 min. at 45° with 4.6 cc. approx. 4N HBr-AcOH gave 0.7 g. NP-Z-L-Ala-DL-NHPh-*OEt*·HBr (XIII·HBr), m. 206-11°. **absolute** EtOH. XIII·HBr (0.7 g.) in 4 cc. DMF treated with 1.2 g. Et<sub>3</sub>N and then with 0.7 g. Z-L-Asp-ONP in 4 cc. THF gave 1.2 g. NP-Z-L-Asp-L-Ala-DL-NHPh-*OEt*. To 1.4 g. Z-Gly in 4 cc. EtOH added 0.025 g. Et<sub>3</sub>N at -10° to -15° until pH 7 was attained, followed during 15 min. by 0.1 g. ClCO<sub>2</sub>Et, the solution stirred approx. 1 min. at -5°, treated with a precooled solution of 2.0 g. X in 12.5 cc. THF at -10°, stirred 30 min. at -5°, and refrigerated 3 days at 0° to give 0.5 g. Z-Gly-NHPhCH(CH<sub>2</sub>Ph)COX (XIV) (*R* = Z-Gly, X = *OEt*) (XV). To 2 g. X and 1.04 g. Z-Gly in 30 cc. MeCN was added 1.23 g. DCCl with stirring and ice cooling and the solution let stand 20 hrs. at 0°, to give 0.4 g. XV. To 3.49 g. X in 20 cc. **absolute** C<sub>5</sub>H<sub>5</sub>N were added simultaneously 2.2 g. *p*-tosyl chloride and 1.66 cc. Et<sub>3</sub>N with ice cooling to give 4.6 g. XIV (*R* = *p*-tosyl, X = *OEt*) (XVI). XVI (1.2 g.) dissolved in 1 cc. **absolute** MeOH by heating, the solution cooled, treated with approx. 100 mg. 1,2,4-triazole and 0.76 cc. 100% N<sub>2</sub>H<sub>4</sub>·H<sub>2</sub>O, and let stand 4 days at room temperature gave 0.1 g. XIV (*R* = *p*-tosyl, X = N<sub>2</sub>H<sub>3</sub>). The mixed anhydride from 1.0 g. Z-Gly and 1.8 g. ClCO<sub>2</sub>Et treated with a precooled solution of 3.0 g. XI in 25 cc. THF as described for XV gave 1.2 g. crude NP-BCCα-(Z-Gly)-DL-NHPh-*OEt* (XVII). NP-tert-butyloxycarbonyl-L-α-hydrazinyl-β-phenylpropionyl amine acid esters was prepared as follows: Method A. To 5 millimoles appropriate amino acid ester-HCl in 4 cc. DMF was added 0.7 cc. Et<sub>3</sub>N with stirring and ice cooling, precipitated Et<sub>3</sub>N·HCl filtered and washed with 1 cc. Et<sub>3</sub>N, the filtrate added to a solution of 1.4 g. III in 10 cc. THF, 1.1 g. DCCl added at 1° and the solution kept approx. 60 hrs. at 0° to give the corresponding heterodipeptide ester. Method B. A solution of 5 millimoles amino acid ester (prepared as in Method A) combined with a solution of 1.88 g. VIIIa in 10 cc. THF, and kept approx. 60 hrs. at 20-25° gave 90-100% corresponding heterodipeptide ester. Thus, with Gly-*OEt*, there was obtained 90% by method A) and 100% by method B) NP-BCC-GL-NHPh-Gly-*OEt* (XVIII). From L-Leu-*Me* was obtained 70% (method A) and 80% (method B) diastereoisomeric mixture of NP-BCC-GL-NHPh-L-Leu-*Me*. L-Ile-Gly-L-Leu-L-Met-NH<sub>2</sub> (Luebke, et al., 1962, 4113) 0.107 g., 1 millimole Et<sub>3</sub>N, and 1 millimole VIIIa in 4 cc. DMF let stand 60 hrs. at 20-25° and diluted with H<sub>2</sub>O gave 0.62 g. NP-BCC-GL-NHPh-L-Ile-Gly-L-Leu-L-Met-NH<sub>2</sub>. A solution of 3.3 millimoles L-Ile-*OMe* (prepared as in method A) combined with a solution of 1.4 g. XI (*R* = OH) in 6 cc. DMF, and treated further like method A gave 0.3 g. NP-[Z-L-Ala-DL-NHPh-L-*OMe*]. XVIII (1.92 g.) in 20 cc. MeOH combined with a solution of 0.2 g. NaOH in 50 cc. H<sub>2</sub>O, a solution of 1.1 g.

NaOH

in 50 cc. H<sub>2</sub>O and 20 cc. MeOH added dropwise during 2 hrs. while maintaining the pH at 8-9 gave 1.1 g. NP-BCC-GL-NHPh-Gly-*Me* (XIX). XIX (0.91 g.) in 10 cc. THF treated first with 0.1 g. *p*-TosCl and then with 0.52 g. DCCl overnight at 0°, gave 1.48 g.

N $\beta$ -BDC-DL-NHPhe-Gly-OEt, m. 90-2°.

IT 14381-16-9P 14381-17-0P

RL: SPN (Synthetic preparation); PPEP (Preparation)  
(preparation of)

119 ANSWER 9 OF 10 HCAPLUS COPYRIGHT 2002 ACS

ACCESSION NUMBER: 1965:438638 HCAPLUS  
DOCUMENT NUMBER: 65:38638  
ORIGINAL REFERENCE NO.: 63:6854h, 6855a  
TITLE: Synthesis of 1,3-bis[bis(carboxymethyl)amino]ethane  
AUTHOR(S): Ermakova, M. I.; Podgornaya, I. V.; Krasova, N. V.;  
Ponovskii, I. Ya.  
CORPORATE SOURCE: Chem. Inst., Sverdlovsk  
SOURCE: Zh. Organ. Khim. (1965), 1(5), 857-60  
DOCUMENT TYPE: Journal  
LANGUAGE: Russian

AB (MeO<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>NNH<sub>2</sub>.HCl treated with aqueous NaOH gave the free ester, b<sub>8</sub> 124-5°, n<sub>D</sub> 1.4562, d<sub>4</sub> 1.1930 [p-nitrobenzylidene derivative m. 75-7°; hydrazone with p,N-bis(β-chloroethyl)aminobenzaldehyde m. 76-8°; picrate m. 154-3°]. This kept 3-4 hrs. in EtOH-NH<sub>3</sub> with CS<sub>2</sub> gave 48% (MeO<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>NNHCS<sub>2</sub>NH<sub>4</sub>, m. 102-4°, which adjusted to pH 3 with HCl gave the free acid, m. 82-4°, unstable in storage. This heated in **absolute** EtOH 50 min. gave SC[NH(CH<sub>2</sub>CO<sub>2</sub>Me)<sub>2</sub>]<sub>2</sub>, m. 88-8°, which refluxed 1 hr. in 10% HCl gave 33% SC[NH(CH<sub>2</sub>CO<sub>2</sub>H)<sub>2</sub>]<sub>2</sub>, decomposes 190-3°. The polarograms of the salts of this acid with 13 common metal ions were reported. This acid in weakly basic medium can complex many metals such as Fe, Co, Ni, Mn, Cr(IV), and Cd. The complex forming tendency is weaker in acid media.

IT 2215-00-1, Acetic acid, [thiocarbonyl]dihydropyrazinylidene diacetate-  
(preparation and polarography of its metal complexes

IT 2509-12-8, Acetic acid, [thiocarbonyl]dihydropyrazinylidene diacetate-  
tetramethyl ester  
preparation of)

119 ANSWER 10 OF 10 HCAPLUS COPYRIGHT 2002 ACS

ACCESSION NUMBER: 1964:469149 HCAPLUS  
DOCUMENT NUMBER: 61:69149  
ORIGINAL REFERENCE NO.: 61:11998a-h, 11999a-h, 12000a-h, 12001a-h, 12002a-h  
TITLE: Synthesis of nitrogen-containing heterocycles. XVII.  
α-Chloro oximes. 2  
AUTHOR(S): Dornow, Alfred; Marquardt, Hans Heinrich; Paucksch,  
Heinrich  
CORPORATE SOURCE: Tech. Hochschule, Hannover, Germany  
SOURCE: Ber. (1961), 97(8), 2105-8  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

GI For diagram(s), see printed CA Issue.

AB cf. CA 55, 2669h, 8386J. α-Chloro oximes with R<sub>1</sub>(SCH<sub>3</sub>)<sub>2</sub> (I) gave 2-aminothiazole 3-oxides (II). The 4-Me 3-ox. of II with 2,4,6-trimethyl-2-amino-4-hydroxyethyl- and 4-chloromethylthiazoles, α-Chloro oximes with EtOCS<sub>2</sub>K gave α-ethoxythioaralkylthio oximes which were cyclized to 2-mercaptothiazole 3-oxides. R<sub>1</sub>CHClCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Cl (III) (R = Ph) in 20 cc. EtOH refluxed 1 hr. with 2.8 g. I in 15 cc. EtOH yielded 3.3 g. IV (R = Ph), m. 181° (H<sub>2</sub>O). p-ClC<sub>6</sub>H<sub>4</sub>CHClCH<sub>2</sub>CH<sub>2</sub>Cl (4.1 g.) in 30 cc. EtOH treated 2 days at room temperature with 2.8 g. I in 15 cc. EtOH yielded

4.6 g. IV (R = p-ClC<sub>6</sub>H<sub>4</sub>) (V), m. 208° (EtOH). IV (R = Ph) (1 g.) in 25 cc. 2N HCl heated 0.5 hr. on a water bath with 2 g. Zn dust yielded 0.5 g. 2-amino-4-methyl-5-phenylthiazole (VI), m. 163° (aqueous MeOH). V (2 g.) in 20 cc. (CH<sub>2</sub>Cl)<sub>2</sub> treated at room temperature with 1.6 cc. AcCl yielded

2.4 g. 2-amino-3-acetoxy-4-methyl-5(p-chlorophenyl)thiazolium chloride, m. 130° (decomposition). V (2 g.) in 150 cc. (CH<sub>2</sub>Cl)<sub>2</sub> refluxed 1 hr. with

1.4 g. AcCl gave 2.6 g. 2-amino-4-methyl-5-(p-chlorophenyl)-1,3,4-thiazole, m. 266° (decomposition) (C<sub>6</sub>H<sub>4</sub>), and 1.4 g. 2-amino-4-hydroxymethyl-5-(p-chlorophenyl)thiazole, m. 190° (C<sub>6</sub>H<sub>4</sub>-EtOH). EtOCS<sub>2</sub>K (3.2 g.) in 25 cc. EtOH added to 3.7 g. III in 25 cc. EtOH and poured after 5 hrs. into 400 cc. H<sub>2</sub>O yielded 4.4 g. 1-ethoxythiocarbonylthio-2-oximino-1-phenylpropane (VII), m. 125° (aqueous EtOH). VII (3.5 g.) and 20 cc. 2N NaOH heated 20 min. at 100° gave 2 g. 1-SH analog (VIII) of IV (R = Ph), m. 143° (MeOH). VIII (0.6 g.), 5 cc. HI (d. 1.9), and 0.3 g. red P refluxed 20 min. yielded 0.4 g. 1-SH analog of VI, m. 151° (MeOH). XXVI. Use of α-amino oximes in the preparation of imidazole 3-oxides. Alfred Lornow and Hans Heinrich Marquardt. Ibid. 1169-72. α-Amino oximes react with ClCO<sub>2</sub>Et (I) and ClCS<sub>2</sub>Et (II) on the NH<sub>2</sub> group to yield the corresponding urethans and thiourethans, resp. The free carbamyl radicals, obtained by alkaline saponification of the urethans and thiourethans, eliminate CO<sub>2</sub> and COS, resp., to yield with cyclization imidazole 3-oxides. MeCl:MeOH:PhNH<sub>2</sub> (1:1 g.) in 30 cc. C<sub>6</sub>H<sub>6</sub> treated at room temperature for 10 hrs. with stirring with 0.4 g. I in 10 cc. C<sub>6</sub>H<sub>6</sub> gave 0.8 g. EtO<sub>2</sub>CNHCH<sub>2</sub>CO<sub>2</sub>Et (III), m. 113° (petr. ether-C<sub>6</sub>H<sub>6</sub>). III (0.3 g.) in 10 cc. 1N NaOH refluxed gave 0.5 g. 3-hydroxy-4,5,5-trimethylimidazole 3-oxide, m. 233° (H<sub>2</sub>O). AcCl<sub>2</sub> (0.15 g.) in 1 cc. 6N HCl added to 3 g. C in 50 cc. H<sub>2</sub>O, and the mixture saturated with H<sub>2</sub> gave the hydrogenation catalyst. MeOH was stirred under MeOH. AcPhCHNH<sub>2</sub> (6.3 g.) in 80 cc. **absolute** MeOH and 1 cc. 10% HCl-MeOH hydrogenated at room temperature over 1.5 g. catalyst yielded 8.4 g. AcPhCHNH<sub>2</sub>.HCl (IV), m. 231° (decomposition). IV (3.3 g.) and 7 g. NH<sub>2</sub>OH.HCl in 30 cc. H<sub>2</sub>O treated rapidly with stirring with 16.5 g. Ac<sub>2</sub>O in 60 cc. H<sub>2</sub>O (heated to 100°) gave 10.1 g. PhCH(NH<sub>2</sub>)CMe<sub>2</sub>CO<sub>2</sub>Et, m. 167° (ist-PrOH), which in 50 cc. H<sub>2</sub>O treated with 1.5 g. Na<sub>2</sub>CO<sub>3</sub> in 15 cc. H<sub>2</sub>O and extracted with CHCl<sub>3</sub> yielded 6.7 g. PhCH(NH<sub>2</sub>)CMe<sub>2</sub>CO<sub>2</sub>Et (V), m. 74° (CHCl<sub>3</sub>-petr. ether), 76° (MeOH). V (3.3 g.) in 150 cc. C<sub>6</sub>H<sub>6</sub> treated slowly with stirring with 1.1 g. I in 20 cc. C<sub>6</sub>H<sub>6</sub> yielded 1.1 g. EtO<sub>2</sub>CNHCH<sub>2</sub>PhCMe<sub>2</sub>CO<sub>2</sub>Et, m. 138° (C<sub>6</sub>H<sub>6</sub>-petr. ether), which heated 10 min. on a water bath with 10 cc. 2N NaOH gave 1.1 g. VI, m. 122° (EtOH). VI (0.6 g.) in 30 g. 80% AcOH refluxed 3 hrs. on a water bath with 4 g. Zn dust gave 0.4 g. 3-hydroxy-4-methyl-5-phenylthiazole, m. 285° (aqueous EtOH). V (3.22 g.) in 150 cc. C<sub>6</sub>H<sub>6</sub> treated slowly with stirring with 1.24 g. II in 30 cc. C<sub>6</sub>H<sub>6</sub>, stirred 1 hr., filtered from the HCl salt, m. 216°, and evaporated, and the viscous, yellow residue heated 4 hrs. on a water bath with 10 cc. 1N NaOH yielded 1.1 g. 1-SH analog of VI, m. 211° (decomposition). aqueous MeOH. XXVII. 1,2,4-Triazines. I. Preparation of some new s-triazolo[3,2-c]-as-triazines. Alfred Lornow, Herbert Mempel, and Hans Marquardt. 1173-3. s-triazolo[3,2-c]-as-triazines (I) with α-oxo acids gave 4-amino-3-oxo-3,4,5-tetrahydro-as-triazines (II) which formed, via the corresponding MeS comp., with amines 3,4-diamino-4,5-dihydroas-triazines (III). III were converted readily with HCO<sub>2</sub>H or Ac<sub>2</sub>O into 3,7-dihydro-s-triazolo[3,2-c]-as-triazines (IV). I (53 g.) in 500 cc. boiling H<sub>2</sub>O treated slowly with 44 g. AcCO<sub>2</sub>H and kept 3 hrs. at room temperature yielded 75 g. II (R = Me) (V, m. 186° (H<sub>2</sub>O). I (1.06 g.) in 50 cc. boiling H<sub>2</sub>O with 1.5 g. HCO<sub>2</sub>H gave 2.1 g. II (R = Ph) (VI), decomposition 161° (H<sub>2</sub>O). V (1 g.) in 1 cc. boiling MeOH treated with 1 cc. Et<sub>3</sub>NH and refluxed 0.5 hr. yielded 1.3 g. 4-PhCH<sub>2</sub>N analog of V, m. 204-6° (C<sub>6</sub>H<sub>6</sub>). V (1 g.) in 10 cc. Et<sub>3</sub>NH treated 3 hrs. with 1 cc. AcCl gave 0.6 g. di-Ac derivative of V, m. 162° (C<sub>6</sub>H<sub>6</sub>). V (3.1 g.) and 1.6 g. NaOH in 30 cc. H<sub>2</sub>O stirred 6 hrs. with 1.3 cc. MeI yielded 2.7 g. H<sub>2</sub>NNHC(SMe):NN:CO<sub>2</sub>Et (VII), m. 145-55° (aqueous MeOH). VII (1.4 g.) in 70 cc. MeOH refluxed 3 hrs. gave 1.7 g. VIII (R = Me) (IX, m. 165° (MeOH). V (15.7 g.) and NaOMe from 2.3 g. Na and 100 cc. **absolute** MeOH refluxed 3.5 hrs. with 2.3 cc. MeI yielded 14 g. IX, m. 166° (H<sub>2</sub>O). VI (22 g.) and NaOMe from 2.3 g. Na and 100 cc. **absolute** MeOH treated during 10 min. dropwise with 13 g. MeI, refluxed

0.5 hr., and kept 12 hrs. at 17° yielded 23 g. VIII (R = Ph, XI), m. 196° (MeOH). IX (1 g.) and 10 cc. BuNH<sub>2</sub> refluxed 3 hrs. yielded 1.1 g. III (R = Me, R<sub>1</sub> = Bu), m. 182° (MeOH); HCOCH<sub>3</sub>-para, m.p. 111-112° (MeOH) and 1 cc. BuNH<sub>2</sub> heated 5 hrs. at 170° gave 0.1 g. III (R = Me, R<sub>1</sub> = Ph), m. 235-6° (MeOH). IX (1 g.) with 4 cc. EtCH<sub>2</sub>NH<sub>2</sub> yielded similarly 1.3 g. III (R = Me, R<sub>1</sub> = EtCH<sub>2</sub>) (XI), m. 167° (aqueous MeOH). X (1.17 g.) and 15 cc. BuNH<sub>2</sub> refluxed 3 hrs. gave 1.9 g. III (R = Me, R<sub>1</sub> = Bu) (XII), m. 142° (MeOH). X (2.34 g.) and 1 cc. BuNH<sub>2</sub> refluxed 1 hr. yielded 1.37 g. III (R = Me, R<sub>1</sub> = EtCH<sub>2</sub>) (XIII), m. 175° (MeOH). X (1 g.) and 5 cc. morpholine heated 2 hrs. at 110° yielded 0.9 g. 4-amino-3-morpholino-5-oxo-6-phenyl-4,5-dihydro-1,2,4-triazine, m. 163° (MeOH). X (4 g.) in 20 cc. BuNH<sub>2</sub> heated 4 hrs. at 150° yielded 1.5 g. III (R = R<sub>1</sub> = Ph) (XIV), m. 211.5° (MeOH). IX (1 g.) and 1.5 g. 95% N<sub>2</sub>H<sub>4</sub> in 30 cc. **abs** iso-PrOH refluxed 4 hrs. gave 0.85 g. III (R = Me, R<sub>1</sub> = NH<sub>2</sub>), m. 283-4° (MeOH). [EtSO<sub>2</sub>(NH<sub>2</sub>):NHNH<sub>2</sub>]Br (100 g.) in 250 cc. H<sub>2</sub>O treated 24 hrs. at room temperature with 35 cc. 30% N<sub>2</sub>H<sub>4</sub>.H<sub>2</sub>O yielded 66 g. HNHC(NHNH<sub>2</sub>)<sub>2</sub>SBr (XVI), m. 159° (MeOH). XV (17 g.) in 100 cc. H<sub>2</sub>O heated 15 min. at 30° with 2 cc. AcOCH<sub>3</sub> yielded 12.4 g. III (R = Me, R<sub>1</sub> = H) (XVI), m. 145° (H<sub>2</sub>O). XV (4.3 g.) in a little H<sub>2</sub>O with 3.8 g. EtCOCH<sub>3</sub> in MeOH heated briefly gave 2 g. III (R = Ph, R<sub>1</sub> = H) (XVII), m. 133-34° (decomposition). XVI (1 g.) and 3 cc. 99% HCOCH<sub>3</sub> refluxed 4 hrs. yielded 0.45 g. IV (R = Me, R<sub>1</sub> = R<sub>2</sub> = H), m. 250-1° (H<sub>2</sub>O). XVI (1 g.) and 5 cc. Ac<sub>2</sub>O refluxed 3 hrs. yielded 0.7 g. IV (R = R<sub>1</sub> = Me, R<sub>2</sub> = H), m. 160-3°. XI (0.5 g.) and 1 cc. HCOCH<sub>3</sub> refluxed 2 min. yielded 0.5 g. IV (R = Me, R<sub>1</sub> = EtCH<sub>2</sub>, R<sub>2</sub> = H), m. 192° (H<sub>2</sub>O). XII (1 g.) in 10 cc. HCOCH<sub>3</sub> refluxed 48 hrs. gave 1.2 g. IV (R = Ph, R<sub>1</sub> = Bu, R<sub>2</sub> = H), m. 166° (MeOH). XIII (0.5 g.) gave similarly 0.1 g. IV (R = Ph, R<sub>2</sub> = H), m. 212° (iso-PrOH). XIII (0.5 g.) and 1 cc. HCOCH<sub>3</sub> refluxed 3 hrs. yielded 0.4 g. IV (R = Ph, R<sub>1</sub> = EtCH<sub>2</sub>, R<sub>2</sub> = H), m. 181-1° (MeOH). XVII (0.5 g.) yielded similarly with 5 cc. Ac<sub>2</sub>O 0.45 g. IV (R = Ph, R<sub>1</sub> = H, R<sub>2</sub> = Me), m. 247-8°. XVI (1 g.) in 60 cc. MeOH refluxed with 1.4 g. EtOCH<sub>3</sub>Br yielded 0.7 g. XVIII, m. 200-2° (HCONMe<sub>2</sub>). XIV (0.5 g.) and 5 cc. Ac<sub>2</sub>O heated 1 hr. at 131° yielded 0.7 g. solid, C<sub>12</sub>H<sub>8</sub>N<sub>4</sub>O<sub>2</sub>, m. 214° (MeOH), presumably a pyrazolo-as-triazine, and 1 g. light yellow prisms, C<sub>12</sub>H<sub>8</sub>N<sub>4</sub>O<sub>2</sub>, m. 123°, a di-Ac compound XXVIII. 1,2,4-Triazines. 2. Preparation of some new s-triazolo[4,3-b]-astriazines. Alfred Dornow, Weiner Abstele, and Herbert Menzel. Ibid. 1179-84. 3-Hydrazino-1,2,4-triazines with CS<sub>2</sub>, urea, or HCCN yielded s-triazolo[4,3-b]-as-triazines. 3-Methylthio-5,6-diphenyl-as-triazine (10 g.) and 10 cc. 80% N<sub>2</sub>H<sub>4</sub>.H<sub>2</sub>O in 200 cc. iso-PrOH refluxed 18 hrs. yielded 16 g. 3-hydrazino-5,6-diphenyl-as-triazine II, m. 170° (MeOH). I and aldehydes or ketones in EtOH refluxed 12 hrs. gave in most cases nearly quant. the 5,6-diphenyl-1,2,4-triazin-3-ylhydrazones of the following compds. (m.p. given): EtCHO, 223° (MeOH); PhCHO, 331° (HCONMe<sub>2</sub>); p-ClC<sub>6</sub>H<sub>4</sub>CHO, 284° (HCONMe<sub>2</sub>); p-MeOC<sub>6</sub>H<sub>4</sub>CHO, 268° (HCONMe<sub>2</sub>); furfural, 333° (EtOH); Me<sub>2</sub>CO, 191° (Me<sub>2</sub>CO); AcPh, 160° (iso-PrOH); EtPh, 111° (iso-PrOH); cyclohexanone, 162° (iso-PrOH). 3-Methylthio-5-oxo-6-methyl-4,5-dihydro-1,2,4-triazine (1.0 g.) in 50 cc. iso-PrOH refluxed 3 hrs. with 15 cc. EtCH<sub>2</sub>NH<sub>2</sub> gave MeNH and 134 g. 5-oxo-3-hydrazino-6-methyl-4,5-dihydro-1,2,4-triazine III, m. 240°. III (1 g.) in 100 cc. boiling MeOH treated with 2.1 g. p-ClC<sub>6</sub>H<sub>4</sub>CHO yielded 0.1 g. 5-oxo-6-methyl-4,5-dihydro-1,2,4-triazin-3-ylhydrazone IV, m. 331° (HCONMe<sub>2</sub>). Similarly was prepared the analogous derivative of p-MeOC<sub>6</sub>H<sub>4</sub>CHO, m. 308° (HCONMe<sub>2</sub>), in 96% yield. IV (R = EtO, R<sub>1</sub> = SH) (11 g.) and NaOEt from 1.5 g. Na and 210 cc. **absolute** EtOH treated 48 hrs. with 8 g. MeI gave 8.5 g. IV (R = EtO, R<sub>1</sub> = MeS) (V), m. 143-3° (H<sub>2</sub>O). IV (R = SH, R<sub>1</sub> = MeS) (11 g.) and 15 cc. concentrated H<sub>2</sub>SO<sub>4</sub> in 400 cc. **absolute** EtOH refluxed 5 hrs. yielded 8.2 g. V, m. 142° (H<sub>2</sub>O). V (2 g.) in 100 cc. iso-PrOH refluxed 3 hrs. with 3 cc. 38% N<sub>2</sub>H<sub>4</sub> yielded 1.4 g. IV (R = R<sub>1</sub> = NHNH<sub>2</sub>), did not melt up to 390° (aqueous MeOH). IV (R = OH, R<sub>1</sub> = SH) (1 g.)

and 6 g.  $\text{ClCH}_2\text{CO}_2\text{H}$  in 60 cc.  $\text{H}_2\text{O}$  refluxed 4 hrs. yielded 1.4 g. VI ( $R_1 = \text{CH}_3$ , decompose  $235^\circ$  (MeOH). I (1.5 g.),  $\text{H}_2\text{O}$  10 cc.  $\text{C}_6\text{H}_5\text{NH}_2$  refluxed 10 hrs. yielded 19.5 g. VII ( $R_1 = \text{Ph}$ ,  $R_2 = \text{Me}$ , VIII, m.p.  $298-300^\circ$  (HCONMe<sub>2</sub>). I,  $\text{C}_6\text{H}_5\text{NH}_2$ , and  $\text{CS}_2$  deposited at room temperature a yellow precipitate, m.  $100^\circ$ , which heated in MeOH decomposed into three components. II (4 g.), 250 cc.  $\text{C}_6\text{H}_5\text{NH}_2$ , and 50 cc.  $\text{CS}_2$  heated at room temp. then heated about 60 hrs. on a water bath until the  $\text{H}_2\text{O}$  evaporated and yielded 5.5 g. VI ( $R_1 = \text{Me}$ ,  $R_2 = \text{CH}_3$ ). VIII, decompose  $312-313^\circ$  (MeOH). 4,5-Diamino-3-thio-2,4-dihydro-1,2,4-triazole (1.31 g.) in 60 cc.  $\text{H}_2\text{O}$  refluxed 5 hrs. with 0.95 g.  $\text{AcCO}_2\text{H}$  yielded 1.2 g. VIII, decompose  $305-31^\circ$  (MeOH). VII (5 g.) in 500 cc. 5% aqueous  $\text{K}_2\text{CO}_3$  treated 6 hrs. with 5 g.  $\text{NaI}$  gave 5 g. IX ( $R_1 = \text{Ph}$ ,  $R_2 = \text{Me}$ ), decompose  $197^\circ$  (MeOH). VIII (3 g.) in 10 cc. 4% aqueous  $\text{NaOH}$  shaken 0.5 hr. with 1.5 cc.  $\text{MeI}$  and adjusted to pH 6 with  $\text{AcOH}$  yielded 2.8 g. IX ( $R_1 = (R_2 = \text{Me}$ ,  $R_1 = \text{CH}_3$ ) (X), m.  $235-7^\circ$  (MeOH). 4,5-Diamino-3-methylthio-1,2,4-triazole (1.45 g.) in 100 cc.  $\text{H}_2\text{O}$  refluxed about 3 hrs. with 0.95 g.  $\text{AcCO}_2\text{H}$  gave 1.31 g. X, m.  $236-3^\circ$  (MeOH). VII (2 g.) and 10 g.  $\text{ClCH}_2\text{CO}_2\text{H}$  in 100 cc. 70%  $\text{AcOH}$  refluxed 4 hrs. gave 1 g. IX ( $R_1 = \text{Ph}$ ,  $R_2 = \text{CH}_2\text{CO}_2\text{H}$ ) (XII), m.  $236^\circ$  (AcOH); Me-ester m.  $161^\circ$  (MeOH). VIII (4 g.) and 40 cc. 10% aqueous  $\text{ClCH}_2\text{CO}_2\text{H}$  refluxed 1 hr. yielded 4.2 g. IX ( $R_1 = \text{Me}$ ,  $R_2 = \text{CH}_2\text{CO}_2\text{H}$ ) (XII), m.  $235^\circ$  (H<sub>2</sub>O). XI (1 g.) in 50 cc. 10% aqueous  $\text{NaOH}$  refluxed 6 hrs. gave 0.5 g. orange-red 3-hydroxy-6,7-diphenyl-5-triazol[4,3-b]-s-triazine (XIII), decompose  $17-18^\circ$  (MeOH). XII (2 g.) and 5 g.  $\text{CS}_2$  heated 10 min. at  $220^\circ$  yielded 0.4 g. XII, decompose  $27^\circ$  (MeOH). I (5 g.) in 15 cc. concentrated  $\text{HCl}$  diluted with 50 cc.  $\text{H}_2\text{O}$  and filtered,

and

the residue dissolved in 1 l.  $\text{H}_2\text{O}$  and treated dropwise with 1.5 g.  $\text{NaOH}$  in 10 cc.  $\text{H}_2\text{O}$  gave 7 g. 3-mercaptoparabono-5,6-diphenyl-2,3-dihydro-as-triazine (XIII), decompose  $205-1^\circ$  (H<sub>2</sub>O). XIII (5 g.) heated 20 min. at  $220^\circ$  yielded 3 g. XII, decompose  $174-80^\circ$  (MeOH). XXIX. 1,2,4-Triazines. 3. Alfred Dornow, Herbert Menzel, and Paul Marx. Ibid. 2185-6. The preparation of I, II ( $R_1 = \text{Me}$ ) (III), II ( $R_1 = \text{H}$ ) (IV), and V is described. 3-Methylthio-5-oxo-1,2,4-dimethyl-2,5-dihydro-1,2,4-triazine (10 g.) in 200 cc. absolute  $\text{EtOH}$  treated 48 hrs. at room temperature with 10 cc. 98%  $\text{H}_2\text{SO}_4$  yielded 0.3 g. 5-oxo-3-hydrazino-2,6-dimethyl-2,5-dihydro-1,2,4-triazine (VI), m.  $241^\circ$  (HCONMe<sub>2</sub>). VI (0.5 g.) in 400 cc. boiling  $\text{MeOH}$  treated with 0.4 g.  $\text{Et}_3\text{N}$  yielded 0.4 g. yellow 5-benzaldehyde analog of VI, m.  $234^\circ$  (MeOH). VI (0.5 g.) and 5 cc.  $\text{HCO}_2\text{H}$  refluxed 3 hrs. yielded 0.2 g. I, m.  $131^\circ$  (H<sub>2</sub>O). VI (1.55 g.) in 10 cc. 2N  $\text{HCl}$  treated dropwise slowly with stirring with 5% aqueous  $\text{NaNH}_2$  yielded 0.7 g. III, m.  $101^\circ$  (C<sub>6</sub>H<sub>6</sub>-petr. ether). 5-Oxo-3-hydrazino-6-phenyl-1,3,4,5-tetrahydro-1,2,4-triazine (VII) (6 g.) in 50 cc. 2N  $\text{HCl}$  gave 3 g.  $\text{NaOH}$  in 10 cc.  $\text{H}_2\text{O}$  gave 4g. IV, m.  $211^\circ$  (MeOH). 5-Hydrazinotetrazole (1 g.) in 10 cc. hot  $\text{H}_2\text{O}$  treated slowly with 0.68 g.  $\text{AcOH}$  gave 1.6 g. the 5-tetrazolylhydrazone (VIII), m.  $211^\circ$  (decomposition) (MeOH), of  $\text{AcOH}$ . VIII (1 g.) and 3 cc.  $\text{AcOH}$  heated to solution and kept 14 hrs. yielded IV, m.  $211^\circ$  (MeOH), and some 5-acetamidotetrazole, m.  $270^\circ$  (decomposition) (AcOH). IV (1.5 g.) treated 12 hrs. at room temperature with 100 cc.  $\text{CH}_3\text{NH}_2$  gave 10 g.  $\text{HCONMeNO}$  yielded 1.1 g. isomer of III, m.  $210^\circ$  (C<sub>6</sub>H<sub>6</sub>). VII (1 g.) in 100 cc. refluxing  $\text{MeOH}$  treated during 5 min. with 5 g.  $\text{Et}_3\text{N}$  and refluxed 3 hrs. gave 0.5 g. 6-methylazeto-phenone 5-oxo-3-hydrazino-6-phenyl-1,3,4,5-tetrahydro-1,2,4-triazine-5-ylhydrazone (IX), m.  $194-5^\circ$  (MeOH). VII (1 g.) in 50 cc.  $\text{MeOH}$  refluxed 1 hr. with 1.2 g.  $\text{EtCH}_2\text{OMe}$  yielded 1.4 g. IX, m.  $196^\circ$  (MeOH). VII (3.5 g.) and 6 g.  $\text{EtCH}_2\text{Br}$  in 10 cc.  $\text{HCONMe}$  heated 1 hrs. at  $100^\circ$  gave 5.2 g. 6-oxo-7-methyl-3-phenyl-4,5-dihydro-4H-as-triazino[4,3-b]-as-triazine, m.  $301^\circ$  (decomposition) (HCONMe<sub>2</sub>). XXX. 1,2,4-Triazines. 4. Preparation of 1,3,4-thiadiazolo[2,3-b]-as-triazines. Alfred Dornow and Paul Marx. Ibid. (9), 2640-6. 3,4-Diamino-1,2,4-triazines and their 3-MeS analogs gave with  $\text{CS}_2$  in  $\text{C}_6\text{H}_5\text{NH}_2$  with the elimination of amine or  $\text{MeSH}$ , resp., the pyridinium salts I. II ( $R_1 = \text{Ph}$ ,  $R_2 = \text{SCH}_3$ ) (X), m.  $210^\circ$  (H<sub>2</sub>O). I (1 g.) in 30 cc. dry  $\text{C}_6\text{H}_5\text{NH}_2$  treated 1 hr. with 15 cc.  $\text{CS}_2$  yielded 6.1 g. I ( $R_1 = \text{Ph}$ ,  $R_2 = \text{H}$ ) (XII), m.  $230^\circ$  (H<sub>2</sub>O). II

(R = Ph, R' = MeS) (5.0 g.), 50 cc. C<sub>5</sub>H<sub>5</sub>N, and 10 cc. CS<sub>2</sub> refluxed 12 hrs. and kept 12 hrs. at room temperature yielded 6.2 g. III. III (5.0 g.) in 20 cc. boiling H<sub>2</sub>O adjusted with concentrated HCl to pH 1 yielded 3.2 g. of product (R = Ph, R' = MeS) (IV), m. 245° (decomposition) (1:1 HCONMe<sub>2</sub>-MeOH). II (R = Me, R' = MeS) (5.0 g.) in 60 cc. dry C<sub>5</sub>H<sub>5</sub>N treated 12 hrs. at room temperature with 10 cc. CS<sub>2</sub> yielded 6.1 g. I (R = Me, R' = MeS) (V), m. 216° (decomposition) (MeOH). II (R = Me, R' = MeS) (5 g.), 40 cc. C<sub>5</sub>H<sub>5</sub>N, and 10 cc. CS<sub>2</sub> refluxed 3 hrs. yielded about 6 g. VI (5.0 g.) in 30 cc. boiling H<sub>2</sub>O adjusted with concentrated HCl to pH 1 gave 3.6 g. IV (R = Me, R' = MeS), m. 240-1° (decomposition) (H<sub>2</sub>O). V in aqueous NaOH heated briefly yielded 1 g. VII (R = Ph) (IX), m. 224° (decomposition) (1:1 aqueous MeOH). 5-Hydroxy-2-thioxo-1,4-thiadiazolidine-HCl (X.HCl) in Et<sub>2</sub>O neutralized with aqueous Na<sub>2</sub>CO<sub>3</sub> and treated with BaCO<sub>3</sub>H gave quant. IX. VII dissolved in dilute aqueous NaOH and acidified gave quant. VIII (R = Me) (XI), m. 116-18° (H<sub>2</sub>O). VII refluxed 1 hr. with dilute HCl gave 100% XI. X in hot H<sub>2</sub>O treated dropwise with BaCO<sub>3</sub>H gave quant. XI, m. 217-19° (H<sub>2</sub>O). IX (3 g.) in 15 cc. AcOH refluxed 5 min. gave 2 g. XII (R = Ph, R' = MeS) (XIII), m. 215° (decomposition) (Ac<sub>2</sub>O). XIII (1.00 g.) in 15 cc. MeOH refluxed 15 min. gave 1.41 g. V, m. 242°. V (1.5 g.) with 1.2 g. Na<sub>2</sub>CO<sub>3</sub> in 7 cc. H<sub>2</sub>O yielded 1.5 g. yellow XIV (R = Ph) (XV), m. 192° (decomposition) (H<sub>2</sub>O). Similarly was prepared the pale yellow XIV (R = Me) (XVI), m. 205° (decomposition) (H<sub>2</sub>O). XV (2.5 g.) in 250 cc. H<sub>2</sub>O treated dropwise with stirring with 1.4 g. MeI and stirred 2 hrs. at room temperature gave 1.5 g. XII (R = Ph, R' = Me, m. 165° (C<sub>6</sub>H<sub>6</sub>-petr. ether). XVI (2.0 g.) and 1.7 g. MeI gave similarly 1.5 g. XII (R = R' = Me, m. 193-6° (H<sub>2</sub>O). V (1.0 g.) and 150 cc. aqueous Na<sub>2</sub>CO<sub>3</sub> heated to 30-35°, treated dropwise with 2.0 g. MeI, and heated 0.5 hr. on water bath gave 1.8 g. XVII (R = Ph, R' = Me, m. 218-19° (AcOH). VII (1.5 g.) in 20 cc. 1% aqueous NaOH shaken with 1.2 g. MeI, kept 1 hr., and adjusted with HCl to pH 3 gave 1.4 g. XVII (R = R' = Me, m. 217-18° (H<sub>2</sub>O). XV (1.0 g.) in 10 cc. HCONMe<sub>2</sub> refluxed 5 min. with 0.5 g. PhCH<sub>2</sub>Cl yielded 1.15 g. XII (R = Ph, R' = PhCH<sub>2</sub>), m. 171° (C<sub>6</sub>H<sub>6</sub>-petr. ether). XVI (1.0 g.) in 6 cc. HCONMe<sub>2</sub> refluxed 1 hr. with 0.7 g. PhCH<sub>2</sub>Cl yielded 1.35 g. XII (R = Me, R' = PhCH<sub>2</sub>), m. 171° (MeOH). VII (1.0 g.) in 10 cc. 10% ClCH<sub>2</sub>CO<sub>2</sub>H refluxed 15 min. gave 1.3 g. XVII (R = Me, R' = CH<sub>2</sub>CO<sub>2</sub>H), m. 219-20° (decomposition) (H<sub>2</sub>O). XV (1.0 g.) in 10 cc. HCONMe<sub>2</sub> refluxed briefly with 0.5 g. ClCH<sub>2</sub>CO<sub>2</sub>H gave 1.85 g. XII (R = Ph, R' = CH<sub>2</sub>CO<sub>2</sub>H) (XVIII), m. 220° (MeOH). XVI (1.1 g.) and 0.5 g. ClCH<sub>2</sub>CO<sub>2</sub>H gave similarly 0.75 g. XII (R = Me, R' = CH<sub>2</sub>CO<sub>2</sub>H), m. 207° (H<sub>2</sub>O). XVIII (1 g.) and 30% aqueous NaOH refluxed 5 hrs. and acidified yielded 1 g. XVII (R = Ph, R' = CH<sub>2</sub>CO<sub>2</sub>H), m. 197° (decomposition) (AcOH). XVI (1.0 g.) in 40 cc. H<sub>2</sub>O treated dropwise with iodine in MeOH until the color persisted gave 0.8 g. XIX, m. 200° (decomposition).

IT 89715-26-4, Pyruvic acid, azine with 3-Me thiocarbamate  
(preparation of)

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E1 THROUGH E16 ASSIGNED

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<http://www.cas.org/ONLINE/STN/STNOTES/stnotes27.pdf>

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L20 ANSWER 1 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 362522-51-8 REGISTRY

CN Hydrazinecarbothioamide, N-[[trans-4-[[[2,5-dioxo-1-pyrrolidinyl]oxy]carbonyl]cyclohexyl]methyl]-, monohydrate  
 (CA INDEX NAME)

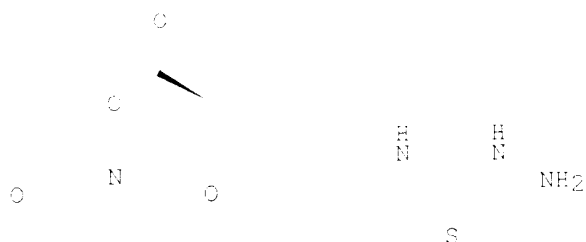
PS STEREOSEARCH

MT D13 H20 N4 O4 S . C1 H

SR CA

LC STN Files: CA, CAPLUS, USPATEFULL

Relative stereochemistry.



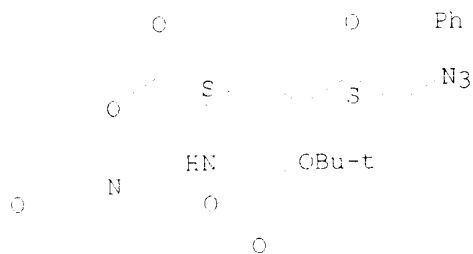
● HCl

1 REFERENCES IN FILE CA (1962 TO DATE)  
1 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 135:272864

L20 ANSWER 2 OF 16 REGISTRY COPYRIGHT 2002 ACS  
RN 144090-64-2 REGISTRY  
CN Carbamic acid, [5-azido-1-[[[(2,5-dioxo-1-pyrrolidinyl)oxycarbonyl]-4-(phenylmethoxy)pentyl]-, 1,1-dimethylethyl ester, [S-(R\*,R\*)]- (9CI) (CA INDEX NAME)  
FS STEREOSEAFCH  
MF C22 H29 N5 O7  
SR CA  
LC STN Files: CA, CAPLUS, CASREACT

Absolute stereochemistry.



1 REFERENCES IN FILE CA (1962 TO DATE)  
1 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 117:212932

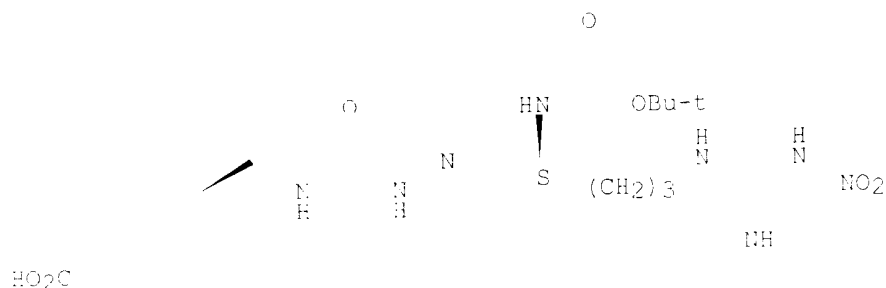
L20 ANSWER 3 OF 16 REGISTRY COPYRIGHT 2002 ACS  
RN 139976-30-0 REGISTRY  
CN Cyclohexanecarboxylic acid, 4-[(7S)-7-[3-[[imino[nitroamino]methyl]amino]propyl]-11,11-dimethyl-3,9-dioxo-10-oxa-2,4,5,8-tetraazadodec-5-en-1-yl]-, trans- (9CI) (CA INDEX NAME)  
OTHER CA INDEX NAMES:  
CN Cyclohexanecarboxylic acid, 4-[(7S)-7-[3-[[imino[nitroamino]methyl]amino]propyl]-11,11-dimethyl-3,9-dioxo-10-oxa-2,4,5,8-tetraazadodec-5-en-1-yl]-, [4(S)-trans]-



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FS STEREOSEARCH  
MF C20 H36 N4 O3  
SR CA  
LN STN Files: BELLESTEIN, CA, CAPLUS, USPATFILL  
\*\*File contains numerically sorted property data

Absolute stereochemistry.  
Double bond geometry unknown.



\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

15 REFERENCES IN FILE CA (1962 TO DATE)  
7 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA  
15 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 132:211416  
REFERENCE 2: 131:229001  
REFERENCE 3: 130:213049  
REFERENCE 4: 127:210992  
REFERENCE 5: 126:131743  
REFERENCE 6: 125:146331  
REFERENCE 7: 124:344120  
REFERENCE 8: 124:176949  
REFERENCE 9: 122:133851  
REFERENCE 10: 121:212001

120 ANSWER 4 OF 16 REGISTRY COPYRIGHT 2002 ACS

KN 139976-29-7 REGISTRY

CN Cyclohexanecarboxylic acid, 4-[[[(hydrazinocarbonyl)amino]methyl]-, trans-,  
mono(trifluoroacetate) (9CI) (CA INDEX NAME)

FS STEREOSEARCH

MF C9 H17 N3 O3 . C2 H F3 O2

SR CA

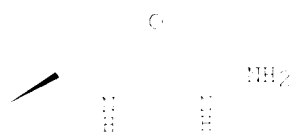
LN STN Files: CA, CAPLUS, USPATFILL

CM 1

CMN 139976-28-6

CMF C9 H17 N3 O3

Relative stereochemistry.



HO2C

CM 2

GRN 76-05-1  
CMF C2 H F3 O2

F

F C CO2H

F

20 REFERENCES IN FILE CA (1962 TO DATE)  
2 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA  
20 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 137:125792  
REFERENCE 2: 137:125391  
REFERENCE 3: 137:125090  
REFERENCE 4: 137:109484  
REFERENCE 5: 137:33541  
REFERENCE 6: 134:281136  
REFERENCE 7: 134:17726  
REFERENCE 8: 133:17829  
REFERENCE 9: 132:251428  
REFERENCE 10: 130:223689

L20 ANSWER 5 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 139976-27-5 REGISTRY

CN Hydrazinecarboxylic acid, 2-[[[(trans-4-carboxycyclohexyl)methyl]amino]methyl]-, 1-(1,1-dimethylethyl) ester [1961] CA INDEX NAME

OTHER CA INDEX NAMES:

CN Hydrazinecarboxylic acid, 2-[[[(4-carboxycyclohexyl)methyl]amino]methyl]-, 1-(1,1-dimethylethyl) ester, trans-

FS STEREOSEARCH

MF C14 H26 N3 O5

SR CA

LC STN Files: BEILSTEIN\*, CA, CAPLUS, USPATFULL

\*File contains numerically searchable property data

Relative stereochemistry.

RUSSEL 147-1147



HO2C

\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

31 REFERENCES IN FILE CA (1962 TO DATE)  
31 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 137:31541  
REFERENCE 2: 134:21136  
REFERENCE 3: 134:115970  
REFERENCE 4: 133:22750  
REFERENCE 5: 132:161428  
REFERENCE 6: 131:129021  
REFERENCE 7: 130:123539  
REFERENCE 8: 128:105143  
REFERENCE 9: 127:146661  
REFERENCE 10: 127:120992

120 ANSWER 6 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 139976-26-4 REGISTRY

CN Hydrazinecarboxylic acid, 2-[[[trans-4-[(phenylmethoxy carbonyl)oxy]-1,1-dimethylethyl ester (9CI) (CA INDEX NAME

OTHER CA INDEX NAMES:

CN Hydrazinecarboxylic acid, 2-[[[4-[(phenylmethoxy carbonyl)oxy]-1,1-dimethylethyl ester, trans-

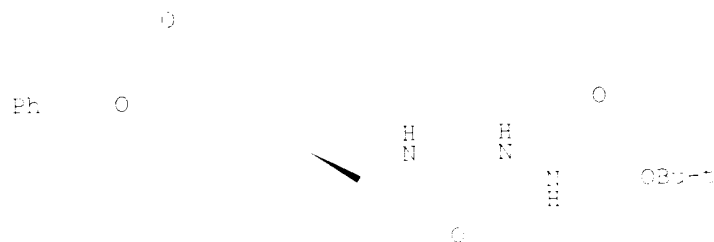
FS STEREOSEARCH

MF C21 H31 N3 O5

SR CA

LC STN Files: BEILSTEIN\*, CA, CAPLUS, USPATFULL  
(\*File contains numerically searchable property data)

Relative stereochemistry.



\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

19 REFERENCES IN FILE CA (1962 TO DATE)  
19 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 131:251426  
REFERENCE 2: 131:229081  
REFERENCE 3: 130:223589  
REFERENCE 4: 130:128401  
REFERENCE 5: 123:205143  
REFERENCE 6: 127:346661  
REFERENCE 7: 127:220992  
REFERENCE 8: 126:131713  
REFERENCE 9: 125:196383  
REFERENCE 10: 124:344129

L20 ANSWER 7 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 134664-50-9 REGISTRY

CN Insulin (cattle-A reduced), N-(2,4-dinitrophenyl)-, tris[2-(hydrazinocarbonyl)hydrazide], 6,7,11,20-tetrakis(hydrogen sulfate) (20)  
(CA INDEX NAME)

OTHER CA INDEX NAMES:

CN Insulin (ox-A reduced), N-(2,4-dinitrophenyl)-, tris[2-(hydrazinocarbonyl)hydrazide], 6,7,11,20-tetrakis(hydrogen sulfate)

FS PROTEIN SEQUENCE; STEREOSEARCH

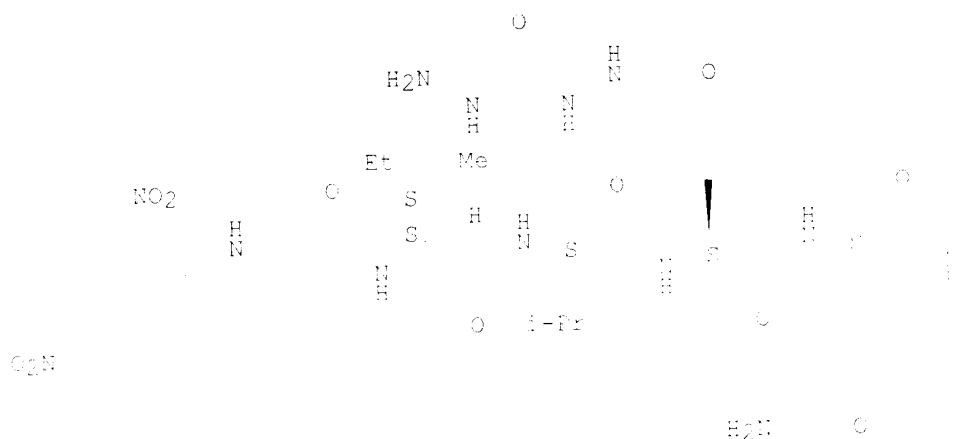
MF C106 H165 N39 O50 S8

SR CA

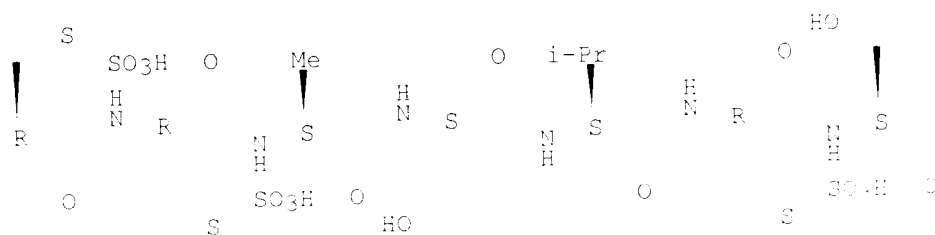
LC STN Files: CA, CAPLUS

Absolute stereochemistry.

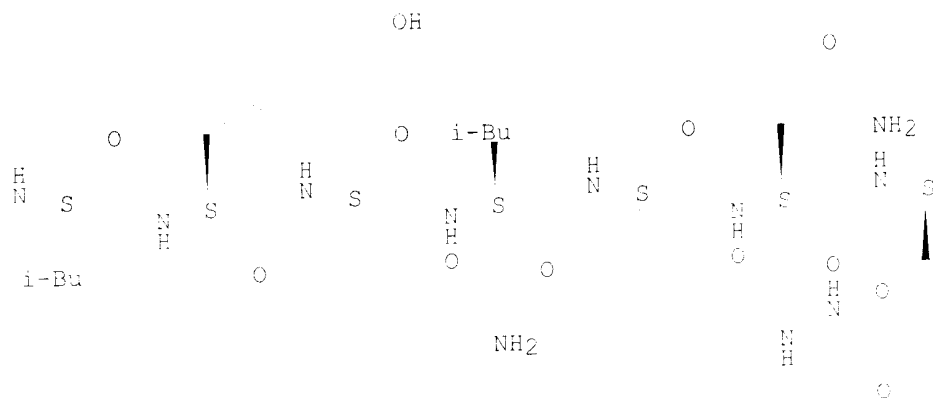
PAGE 1-A



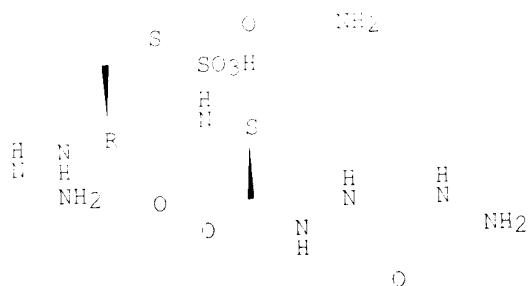
PAGE 1-2



PAGE 1-2



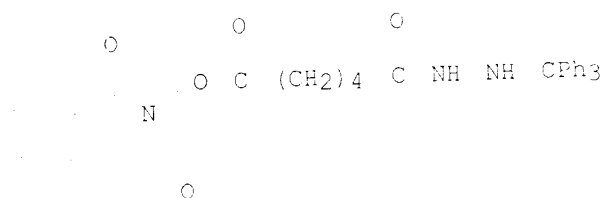
OR



1 REFERENCES IN FILE CA (1962 TO DATE)  
1 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 115:25407

L20 ANSWER 8 OF 16 REGISTRY COPYRIGHT 2002 ACS  
RN **127381-73-1** REGISTRY  
CN Hexanoic acid, 6-[[(1,3-dihydro-1,3-dioxo-2H-isoindol-2-ylidene)-O-methyl-  
2-(triphenylmethyl)hydrazide (9CI) (CA INDEX NAME)  
MF C33 H29 N3 O5  
SR CA  
LC STN Files: CA, CAPLUS, TOXCENTER



\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

1 REFERENCES IN FILE CA (1962 TO DATE)  
1 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 113:2671

L20 ANSWER 9 OF 16 REGISTRY COPYRIGHT 2002 ACS  
RN **89715-26-4** REGISTRY  
CN Pyruvic acid, azine with S-methyl thiocarbamate (9CI) (CA INDEX NAME)  
FS 3D CONCORD  
MF C5 H10 N4 O2 S  
LC STN Files: BEILSTEIN\*, CA, CAOLD, CAPLUS, CASREACT  
(\*File contains numerically searchable property data)

SM-

N N C NH NH<sub>2</sub>Me C CO<sub>2</sub>H

\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

1 REFERENCES IN FILE CA (1962 TO DATE)  
 1 REFERENCES IN FILE CAPLUS (1962 TO DATE)  
 1 REFERENCES IN FILE CAOLD (PRIOR TO 1962)

REFERENCE 1: 61:69149

L20 ANSWER 10 OF 16 REGISTRY COPYRIGHT 2002 ACS  
 RN 50883-75-5 REGISTRY  
 CN Carbonic dihydrazide, (1-methyl-2-oxopropylidene)- (8C1) (CA INDEX NAME)  
 OTHER NAMES:  
 CN (α-Acetyloethylidene)carbohydrazide  
 FS 3D CONCORD  
 MF C5 H10 N4 O2  
 LC STN Files: BEILSTEIN\*, CA, CAPLUS, TOXCENTER  
 (\*File contains numerically searchable property data)

O

H<sub>2</sub>N NH C NH N O

Me C C Me

\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

1 REFERENCES IN FILE CA (1962 TO DATE)  
 1 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 80:48710

L20 ANSWER 11 OF 16 REGISTRY COPYRIGHT 2002 ACS  
 RN 14994-19-5 REGISTRY  
 CN Carbamoyl azide, terephthaloyldi- (8C1) (CA INDEX NAME)  
 FS 3D CONCORD  
 MF C10 H6 N8 O4  
 LC STN Files: CA, CAPLUS

C O

C NH C N3

N3 C NH C

O O

1 REFERENCES IN FILE CA (1962 TO DATE)  
 1 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 66:55437

120 ANSWER 12 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 14381-17-0 REGISTRY

CN Succinimide, N-[[ $\alpha$ -(2-carboxyhydrazino)hydrocinnamoyl]]- $\alpha$ -benzyl ester, DL- (9CI) (CA INDEX NAME)

OTHER CA INDEX NAMES:

CN Hydrocinnamic acid,  $\alpha$ -(2-carboxyhydrazino)-,  $\alpha$ -benzyl ester, O-succinimido deriv., DL-

MF C21 H21 N3 O6

LC STN Files: BEILSTEIN\*, CA, CAPLUS

(\*File contains numerically searchable property data)

O Ph

O O

O N HN O N H O Ph

\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

2 REFERENCES IN FILE CA (1962 TO DATE)

2 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 67:117258

REFERENCE 2: 66:55728

120 ANSWER 13 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 14381-16-9 REGISTRY

CN Succinimide, N-[[ $\alpha$ -(2-carboxyhydrazino)hydrocinnamoyloxy]]-, tert-butyl ester (9CI) (CA INDEX NAME)

OTHER CA INDEX NAMES:

CN Hydrocinnamic acid,  $\alpha$ -(2-carboxyhydrazino)-,  $\alpha$ -tert-butyl ester, O-succinimido deriv., DL-

MF C18 H23 N3 O6

LC STN Files: BEILSTEIN\*, CA, CAPLUS

(\*File contains numerically searchable property data)

O Ph

O O

O N HN O N H O Bu-t

\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*



RUSSEL 09 / -10372

1 REFERENCED IN FILE CA (1962 TO DATE)  
1 REFERENCED IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 66:15725

120 ANSWER 14 OF 16 REGISTRY COPYRIGHT 2002 ACS  
RN 13506-12-2 REGISTRY  
CN Semicarbazide, 4,4'-phthaloylbis[1-phenyl- (8CI) (CA INDEX NAME)  
OTHER NAMES:  
CN Carbamic acid, terephthaloyldi-, bis(2-phenylhydrazide)  
FS 3D CONCORD  
MF C22 H20 N6 O4  
LC STN Files: BEILSTEIN\*, CA, CAPLUS  
(\*File contains numerically searchable property data)

O O

C NH C NH NHPh

PhNH NH C NH C

O O

\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

2 REFERENCES IN FILE CA (1962 TO DATE)  
2 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1: 69:52073

REFERENCE 2: 66:75807

120 ANSWER 15 OF 16 REGISTRY COPYRIGHT 2002 ACS  
RN 2509-12-8 REGISTRY  
CN Acetic acid, (carbonothioyldihydrazinylylidene)tetra-, tetramethyl ester  
(8CI) (CA INDEX NAME)  
OTHER CA INDEX NAMES:  
CN Acetic acid, [(thiocarbonyl)dihydrazinylylidene]tetra-, tetramethyl ester  
(7CI)  
FS 3D CONCORD  
MF C13 H22 N4 O3 S  
LC STN Files: BEILSTEIN\*, CA, CACLD, CAPLUS  
(\*File contains numerically searchable property data)

O

S CH2 C OMe

C NH C NH N CH2 C OMe

MeO C CH2 N CH2 C OMe O

O

\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

RUSSEL 09 / 815976

2 REFERENCES IN FILE CA (1962 TO DATE)  
2 REFERENCES IN FILE CAPLUS (1962 TO DATE)  
1 REFERENCE IN FILE CAOLD (PRIOR TO 1967)

REFERENCE 1: 63:38639

REFERENCE 2: 63:38638

L20 ANSWER 16 OF 16 REGISTRY COPYRIGHT 2002 ACS

RN 2215-00-1 REGISTRY

CN Acetic acid, 2,2',2'',2'''-(carbonethioylidene-1-hydraziny-1-ylidene)tetrakis- (9CI) (CA INDEX NAME)

OTHER CA INDEX NAMES:

CN Acetic acid, [(thiocarbonyl)dihydrazinylylidene]tetra- (7CI, 8CI)

OTHER NAMES:

CN 1,5-Thio-carbohydrazidotetracetic acid

FS 3D CONCORD

MF C9 H14 N4 O8 S

LC STN Files: BEILSTEIN\*, CA, CAOLD, CAPLUS

(\*File contains numerically searchable property data.)

S CH2 CO2H

NH C NH N CH2 CO2H

HO2C CH2 N CH2 CO2H

\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

2 REFERENCES IN FILE CA (1962 TO DATE)  
3 REFERENCES IN FILE CAPLUS (1962 TO DATE)  
1 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

REFERENCE 1: 78:158585

REFERENCE 2: 72:8987

REFERENCE 3: 63:38638